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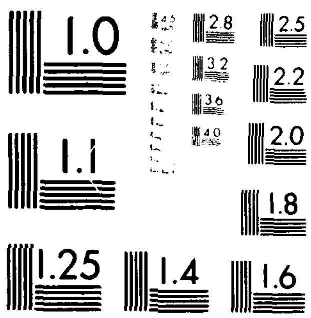

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NONDESTRUCTIVE TESTING OF ARMAMENT-SYSTEM COMPONENTS BY MEANS OF NEUTRON DIFFRACTION

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I. INTRODUCTION

In recent years, increasing emphasis has been placed on nondestructive testing (NDT) techniques for quality control in a large variety of applications. Progress in this area has considerable importance for DOD because of the increasing complexity and cost of armament systems and the inherent limitations of quality control by means of traditional, destructive test methods. This is dramatically illustrated by the on-going, multi-billion dollar DA plant modernization project within which incorporation of state-of-the art NDT methods is an essential part.

Various NDT techniques in which x-rays are employed (e.g. radiography, diffraction) are already well established as quality control methods. The use of neutrons which complements and parallels x-ray techniques has been -- except for neutron radiography -- neglected for NDT, primarily because their use is generally limited to a laboratory environment (e.g. a research reactor). Despite this current limitation, there has been a renewed interest in utilizing neutron scattering for certain technological applications. In

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particular, sizeable programs have been completed at European reactor facilities and begun in the U.S. to construct sophisticated instruments for small-angle neutron scattering.

In the present paper we describe the utilization of wide-angle neutron diffraction for the nondestructive characterization of two properties of importance in metallurgical systems: texture (i.e. preferred grain orientation) and residual stress. The complementary nature of neutron diffraction relative to x-ray diffraction is illustrated by a detailed description of texture measurements for shaped-charge liners and tungsten-alloy kinetic-energy penetrators. The potential of small-angle neutron scattering for nondestructive characterization of precipitates and voids in metals is also discussed.

II. BACKGROUND

A. General (1)

Thermal neutrons from a reactor beam tube are employed for diffraction experiments; typically, neutron velocities of 2 km/sec with an energy of 0.02eV and a wavelength, λ, of 2 A are in the useful range. In contrast to x-rays, thermal neutrons have both energy and wave-vector ($2\pi/\lambda$) in a regime characteristic of lattice vibrations or phonons. Extremely important results relating to interatomic forces have come out of the use of this property in <u>in</u>elastic neutron scattering studies of condensed matter. Microstructural properties, such as texture and residual stress, are probed by elastic scattering (2), to which inelastic scattering contributes only a relatively small background.

Neutron scattering processes can be described by approximating the nucleus as a hard sphere of radius 10^{-12} cm. With x-rays, the atomic scattering factor is a function of scattering angle and is proportional to atomic number. This results from the fact that with x-rays the electrons are the scatterers so that the atom has a finite dimension relative to the x-ray wavelength. This is not the case for elastic scattering of neutrons by the nucleus so that the nuclear scattering power, called scattering length, b, is not angularly dependent (except in the case of magnetic scattering which is outside the scope of the present discussion).

Formally, the intensity measured in a diffraction experiment is proportional to the square of the structure factor $F(\vec{Q})$ where

$$F(\vec{Q}) = \int_V \rho(\vec{r}) e^{i\vec{Q}\cdot\vec{r}} d\vec{r}. \tag{1}$$

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In Eqn. (1), $\rho(\vec{r})$ is the "scattering density", $\vec{Q} = \vec{k}_i - \vec{k}_f$ is the wave-vector transfer, $k_i = 2\pi/\lambda = k_f$, and the integration is taken over the sample volume. In the neutron scattering case

$$\rho(\vec{r}) = \sum b_i \delta(\vec{r}-\vec{r}_i) \tag{2}$$

where b_i is the scattering length of each nucleus in the sample and δ is the Dirac delta function. From Eqns. (1) and (2)

$$F(\vec{Q}) = \sum b_i e^{i\vec{Q}\cdot\vec{r}_i} \tag{3}$$

which gives rise to maxima in the intensity when the Bragg reflection conditions are fulfilled, i.e. when

$$n\lambda = 2d \sin(\theta/2) \tag{4}$$

where d is the plane spacing, θ the scattering angle and n is an integer.

Two important differences between x-ray and neutron scattering processes make the use of neutrons essential for certain applications. As mentioned above, scattering density in the x-ray case varies continuously with atomic number. Scattering density in the neutron case varies discontinuously with atomic number and, in fact, differs for different isotopes of the same element.* In addition, both types of radiation may be absorbed by the scatterers. Typical absorption coefficients, μ, for neutrons and x-rays are on the order of 0.3 and 1000 cm^{-1}, respectively, where intensity after absorption is $I = I_o \exp(-\mu x)$, with I_o the initial intensity and x the sample thickness. For metallurgical samples, neutrons are, in general, on the order of 1000 times more penetrating than x-rays of the same wavelength. In Figure 1, the use of the penetrating power of neutrons for wide-angle diffraction studies of bulk properties in NDT applications is shown schematically.

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*Dramatic illustration of this is found in structural studies of metal hydrides where hydrogen locations are invisible to x-ray but not to neutron diffraction; substitution of deuterium for hydrogen further enhances the sensitivity of the neutron method.

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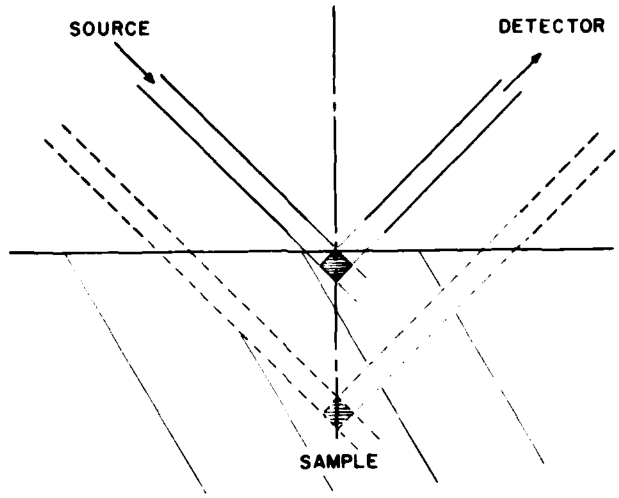

Figure 1. Schematic drawing of stress or texture vs. depth measurement. Heavier dashed and solid lines represent collimators. Lighter lines represent neutron beam path. For single scattering, neutrons reaching the detector can only come from the regions indicated by cross-hatching.

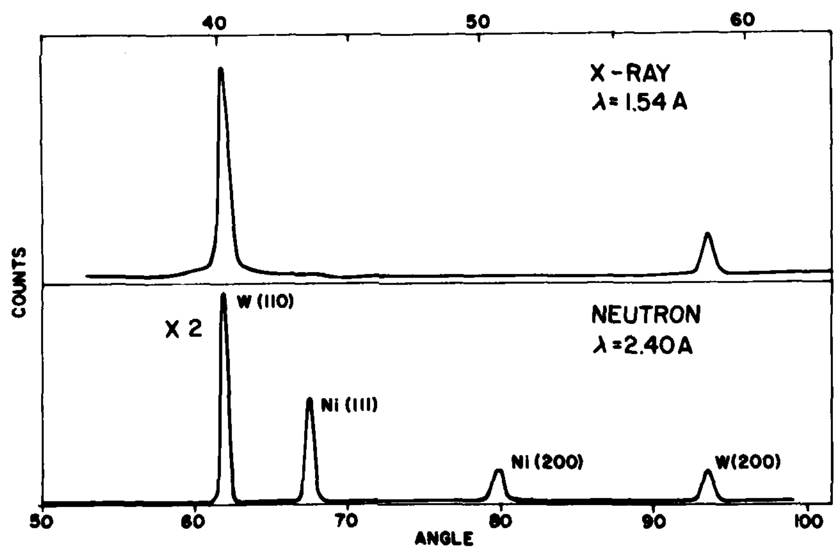

Figure 2. Partial diffraction patterns for a .97 tungsten - .018 nickel - .012 iron (by weight) KE penetrator sample.

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B. Metallurgical properties (3)

1. General: Metal end-items are composed of individual crystallites or grains, the diameters of which may range from 1.00 mm (ASTM No. -3) to 0.006 mm (ASTM No. 12). Although the intrinsic properties of individual grains (e.g. elastic moduli, number and orientation of slip planes) are important in determining the mechanical characteristics of a specimen, the polycrystallinity introduces properties which are often dominant. For example, grain size, intra- and inter-grain boundaries, preferred grain orientation (or texture), dispersion of precipitate particles and residual stresses are interactive (to varying degrees) and affect mechanical properties. In the following subsections three of these properties for which neutron scattering studies have been made are considered in greater detail.

2. Texture: A metal which has undergone severe deformation, as in rolling or swaging, will develop an overall preferred crystallographic orientation or texture. That is, the individual grains tend to orient themselves in a preferred manner with respect to the direction of maximum strain. This arises from the tendency of slip planes in single crystals to rotate parallel to the axis of principal strain. Individual grains are anisotropic but their random aggregation leads to isotropy with respect to mechanical properties. However, preferred alignment of grains introduces anisotropy in mechanical properties which leads to uneven response of the material during forming and fabrication processes and in use.

3. Residual stress: A body which has undergone nonuniform plastic deformation can retain a system of stresses within the body after the external forces have been removed. Macro residual stresses vary continuously through the volume of the body over regions which are large compared with atomic dimensions. They are produced by virtually all forming operations, welding, nonuniform cooling of ingots and electroplating. The important point to be made is that the response of a body in a particular application includes the superposition of the external stress system and the residual stresses. For example, a compressive residual stress (as produced by autofrettage of gun tubes) will reduce the effectiveness of an applied tensile stress in producing fatigue failure, while residual tensile stresses will increase the ease with which failure occurs.

Another type of residual stress, microstress or textural stress, should also be mentioned. These stresses act over dimensions as small as several crystallographic unit cells but may extend throughout a grain. Stress developed around a second-phase precipitate

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is an example of this. Nitriding and carburizing are processes in which microstresses around each particle produce a macro residual stress on the treated surface.

4. Second-phase hardening: Most commercial alloys contain a heterogeneous microstructure consisting of two or more metallurgical phases. The strengthening produced by a fully dispersed insoluble second phase in a metallic matrix is known as dispersion hardening. Precipitation hardening, which also produces strengthening, is achieved by solution treating and quenching an alloy in which a phase in solid solution at elevated temperatures precipitates upon quenching and aging at a lower temperature. Many of the factors which affect strengthening, such as size, shape and number of the second phase particles, have been very difficult to measure with any degree of precision.

The following section describes armament components in which texture, residual stress and precipitation hardening are known to be important for optimizing performance, and the use of neutrons for characterizing these properties.

III. RESULTS AND DISCUSSION

A. Texture

1. Tungsten-alloy KE penetrators: Kinetic-energy penetrator rounds represent a major part of the Army's armor-defeating weapons. With continual improvements in fielded armor, increased penetration is necessary, preferably without increases in size or weight of the rounds. In this subsection we examine one property of state-of-the art tungsten-alloy penetrators: the anisotropy of texture produced by various types of cold working.

The samples are composed of tungsten, nickel and iron in a wt % ratio of 97/1.8/1.2. Sample preparation - which involves isostatic pressing, sintering, heat treatment and cold working - leads to a system consisting of a "tungsten" phase (96.5 wt %) and a "matrix" phase (3.5 wt %). The approximate compositions by weight of the two phases are 99.7W - 0.1Ni - 0.2Fe and 55Ni - 23Fe - 22W, respectively. The potential of neutron diffraction for studies of this type of sample can be inferred from Figure 2 in which partial x-ray and neutron diffraction patterns which cover the same range in lattice spacing are shown. The full diffraction patterns show that the tungsten phase can be indexed according to the bcc tungsten structure with $\underline{a}$ = 3.160(1) A, and the matrix phase can be indexed according to a fcc nickel structure with $\underline{a}$ = 3.586(1) A. For alloys of these compositions and relative concentrations, the matrix phase is

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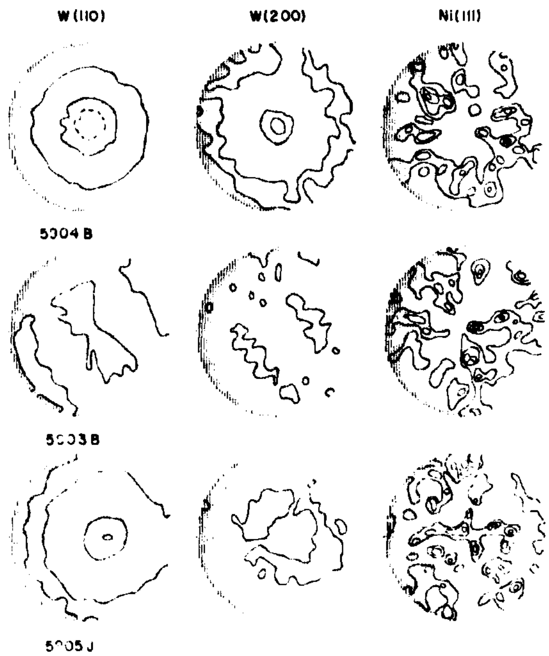

Figure 3. Texture distributions for the three 8% cold-worked tungsten-alloy penetrator samples described in the text. The center of each figure corresponds to the cylinder-axis direction; the periphery of each figure corresponds to a 70% tilt with respect to the cylinder axis. Angular displacement around the periphery corresponds to rotations of the sample about its own axis. Contour lines show "random" and 15% increases in orientation with respect to random (dashed contour = decrease).

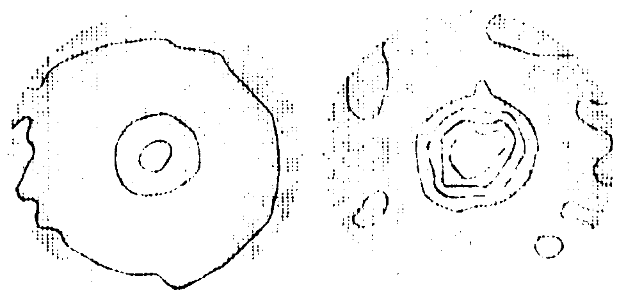

Figure 4. W(110)-on left-and Ni(111) textures for a 16% R.A. swaged sample.

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virtually invisible to x-rays. In contrast, two matrix-phase peaks are clearly visible in the neutron pattern which makes possible texture studies and, in principal, residual stress studies of the matrix phase.

Texture resulting from various types of cold working have been measured for these types of alloys. In Figure 3 are shown pole-figure patterns for one matrix-phase and two tungsten-phase reflections. These were obtained for a 1 cm region at the leading or front end of the 0.7 cm diameter, 10 cm long small-scale penetrators. The three sets of pole figures correspond to samples which have been cold worked by "upsetting" with an 8% increase in cross-section (5904), and swaged with an 8% reduction in area (5903 and 5905). Cold working of the latter pair was performed at two different fabrication facilities. As one would expect, the fiber textures of the "upset" and swaged samples are different. On the other hand, the pattern of sample 5903 is essentially untextured, whereas that of 5904 shows definite texture in the tungsten phase for nominally, the same cold working. The matrix phase (111) pattern shows little if any clear texture.*

In contrast to the above results, samples which have been swaged to a 16% reduction in area show very pronounced tungsten <u>and</u> matrix phase textures (Figure 4). The high degree of axial orientation of the (111) slip planes in the 16% R. A. penetrators relative to the 8% cold-worked samples suggests the possibility of quite a different response to axial stresses in the two types. Although much less pronounced, there is some indication of differences in (111) slip plane texture among the three 8% cold-worked samples. Since the matrix phase is the binder which provides cohesion and strength to the penetrators, test-firing of inspected penetrators -- which is possible because the neutron measurements are truly nondestructive -- is imperative to further test these possibilities.

2. Metallurgically-compensated shaped-charge liners: The second major type of armor-defeating round employed by the Army utilizes the shaped-charge principle. A typical shaped-charge missile has a steel body fitted at the forward end with a cone-shaped metal liner (as shown in Figure 5) whose apex extends backward into the body cavity. On impact, the detonation wave of the charge collapes the

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*A pole figure is a map of the orientational distribution of the normal direction to a specific crystallographic plane (2). The diffractometer geometry is fixed to observe scattered radiation for the Bragg reflection of interest, and the sample rotated - over as large an angular range as possible - about two mutually perpendicular axes. The pole figure is a two-dimensional map of intensity maxima and minima as a function of sample orientation.

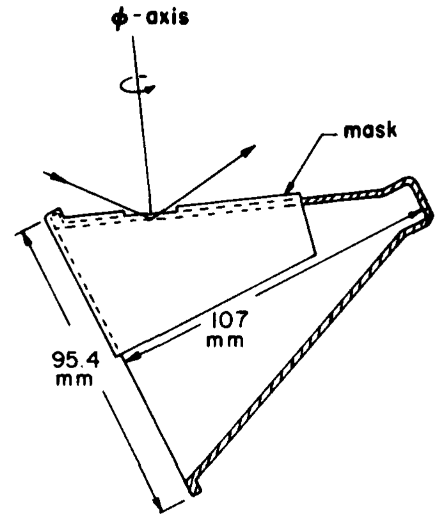

Figure 5. Shaped-charge liner and scattering geometry for neutron diffraction texture measurement.

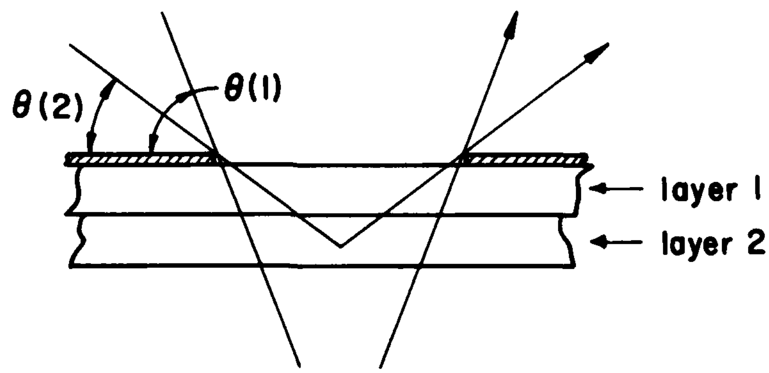

Figure 6. Schematic illustration of sample volume vs. Bragg angle for two-layer, thin sample.

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cone from apex to base sequentially and produces a long narrow jet of cone material traveling along the cone axis with extremely high speed. This jet, which is solely responsible for producing the deep hole in armor, breaks up prematurely when the projectile is rotating about its axis at impact. "Spin compensation" is the term applied to methods employed to counteract degradation of the jet because of rotation.

Spin compensation can be achieved when the cone-shaped metal liner is manufactured by the rotary extrusion process. It has been shown by x-ray diffraction (5) that the critical parameters in this case are related to the alignment of slip planes and slip directions in the cone. However, a quantitative correlation between these metallurgical properties and spin compensation (or performance) has never been established because of the lack of a nondestructive technique for determination of texture.

At the 1976 Army Science Conference we demonstrated that neutron diffraction could be used to nondestructively determine the average texture within the liner wall (6). However, the desired nondestructive determination of texture as a function of depth in the liner wall did not appear possible (by, for example, the technique shown in Figure 1) because the wall was too thin relative to minimal neutron beam dimensions.

Since then we have conceived and tested an approach by which the needed measurement becomes feasible (7). The method makes use of the fact that for an appropriately masked sample, as shown in Figure 6, different depths are probed for a given set of crystallographic planes by changing the Bragg angle. With reference to Eqn. (4), this is achieved either by changing wavelength (which is continuously variable in the neutron case) or by examining higher orders of a given reflection (e.g. 220 or 330 and 110). For a two layer sample, as shown in Figure 6, in the $\theta(2)$ Bragg-angle configuration a volume is examined which is almost entirely layer 1; the $\theta(1)$ configuration includes layer 1 and layer 2 almost equally. Since the overall geometry can be measured to very high precision, appropriately weighted differences of scattered intensities allow separation of the layer 1 and layer 2 textures. The method has been tested with striking success for a two-plate, flat Cu sample (7). The (200) plane texture for each 0.8 mm thick plate was extracted from (200) and (400) texture distributions of the two plates together.

The method has now also been applied to the principal problem of interest: texture as a function of depth for a metallurgically compensated 105 mm HEAT-T liner. Measurements were made employing a diffractometer with fixed wavelength (1.26 A) so that only limited characterization was possible. Nevertheless, at a position 25.4 mm above the base (see Figure 5), the average orientation of the (111) slip planes within two layers was obtained. In Figure 7 texture distributions are shown for (111) and (100) reflections for the total

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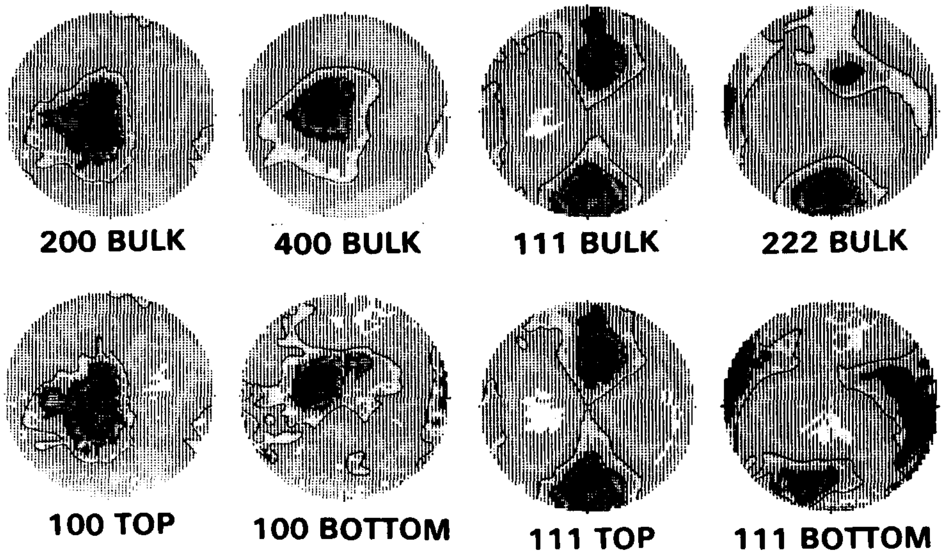

Figure 7. Textures for shaped-charge liner obtained as described in the text. The center of each figure corresponds to the normal direction to the cone surface. The cone-apex direction is toward the top of the page. Other details as in caption, Figure 3.

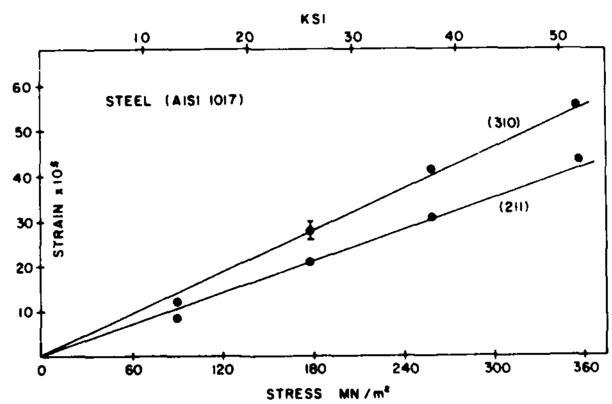

Figure 8. Radial strain vs. stress for a 1.59 cm diameter steel rod in tension.

thickness and the extracted average textures for the 0.5 mm thick "surface" layer and the 2.3 mm thick lower layer. As one would expect, the rotary extrusion fabrication process produces a texture gradient across the thickness of the liner. Our technique and measurements are the first nondestructive characterization of these texture gradients which, when pursued under optimized conditions, have the potential for providing the first texture gradient vs. performance correlation for metallurgically compensated shaped-charge munitions.

B. Residual Stress

Macro residual stresses have long been recognized to be of great importance in determining the lifetime and performance of armament-system components in the field. Programs to measure residual stresses in such diverse items as helicopter rotor blades and hubs, high performance gears, gun tubes, tank track pins and torsion bars, KE penetrators, shaped-charge liners, shell-casings and the general category of weldments are, or have recently been, active. Microstresses are believed to be of less importance than macro residual stresses in hardware and will not be considered here explicitly.

Among nondestructive techniques for measurement of residual stress, x-ray diffraction and ultrasonic methods have received the greatest attention. The former has, in fact, been developed to the point where portable equipment for in-field inspection is available. Because of the limited penetration of x-rays in the energy range suitable for diffraction, this technique is truly nondestructive only for surface measurements. Ultrasonic methods show great promise for characterization of residual stress gradients in the bulk of metallurgical samples. However, the presence of internal interfaces, preferred grain orientation and other inhomogeneities introduce large uncertainties into the measurements.

In principal, neutron diffraction techniques -- as in the case of texture -- exactly parallel x-ray methods for residual stress measurement. The enormously greater penetration of neutrons makes them potentially useful for nondestructive determination of residual stress gradients in the bulk, following the approach illustrated in Figure 1. However, the measurement of residual stress by diffraction methods is much more difficult than texture measurements. The determination consists of measuring strain resulting from residual stress as manifested by the changes in d-spacing and angle of Bragg reflection (see Eqn. 4). Depending on the lattice planes examined and the radiation wavelength, the change in $\theta/2$ is on the order of $0.05°$ in going from unstrained sample to the yield point. Neutron diffraction measurements benefit from the fact that lineshapes of diffracted peaks are Gaussian, which is a considerable aid in analysis. On the other hand, intensities available at even the best of research reactors is less by

orders-of-magnitude than that of analagous x-ray diffraction instruments. This limitation makes the counting time necessary for the measurement of subtle shifts in peak positions impractically long with current "standard" neutron diffractometers. The introduction of focused beams and position-sensitive area detectors would alleviate this difficulty. We have, therefore, concentrated on the more fundamental questions related to the determination of residual stress by neutron diffraction.

Studies which have been completed have established that: 1) no competing effects (e.g. multiple scattering) distort the peak position obtained in the subsurface type of measurement illustrated in Figure 1; 2) the two-dimensional (x-ray) formalism for extraction of stresses from measured strains can be extended to three-dimensions; and 3) because of the Gaussian peak shape the Bragg peak centroid can be determined to on the order of thousandths of a degree even though the resolution is on the order of a degree. The confirmation of the last point is illustrated in Figure 8 in which measured radial strain vs. applied stress is shown for a steel rod in axial tension. The results for two crystallographic planes are shown and the ratio of elastic modulus to Poisson's ratio extracted from the measured slopes are in excellent quantitative agreement with theoretical values for polycrystalline samples reported in the literature (8).

Current work in this area is directed toward the determination of three-dimensional stress gradients in a prototype sample employing a well-collimated beam. Armament components of simple geometry such as KE penetrators would follow.

C. Second-phase Hardening

Small-angle x-ray scattering (SAXS) has been used to study small-concentration precipitates in alloys for decades (9). This approach also makes use of Bragg scattering (Eqn. 4), but in this case scattering which takes place within a few degrees reveals structural properties in the 10 to 1000 A range. With the development of area detectors and new high flux reactors, small-angle neutron scattering (SANS) has in the last few years also been brought to bear on the study of this area of physical metallurgy. Because of the great penetration of the neutron and the different scattering selectivity relative to x-rays, previously unexplorable problem areas now may be amenable to solution, some of them in the context of NDT.

To date, the most interesting SANS work in the area of technological alloys has been done by a group from the FIAT Co. One example of their efforts is the study of the evolution as a function of service time of the γ' precipitate in nickel-base, superalloy UDIMET-710 in turbine blades (10). They clearly observe the accelerated growth of the γ' particles prior to failure.

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At the NBS reactor a state-of-the art SANS instrument is nearing completion. An interim facility has been operating with which a variety of problems have been studied in a preliminary way. In the metallurgical area, SANS has been used to study creep-induced voids in iron (10). Fe-0.2% Ti subjected to a σ = 35 ksi, ϵ = 1.2% stress at 500°C showed voids of 360 A average diameter with SANS but not with SEM. These results illustrate the relative ease by which SANS can characterize metallurgically important defects in the nucleation stage nondestructively.

Upon completion of the new facility, second-phase hardening studies for armament related alloys will be pursued. Among these, the effect of cold work and heat treatment on precipitates in new, tungsten-based, KE penetrator alloys requires immediate characterization.

IV. SUMMARY AND CONCLUSIONS

In the present work we have described the limitations and advantages of neutron diffraction for nondestructive determination of certain properties in metallurgical samples. In the area of residual stress, several necessary tests of the neutron diffraction method have been made and it has been shown for the first time that the technique has the potential for determination of three-dimensional stress gradients in the bulk.

In the area of texture or preferred grain orientation determination, both neutron selectivity and penetration have been used for specific armament system applications. We have conceived and tested a method by which texture can be characterized nondestructively in samples which are thin relative to typical neutron diffraction beam dimensions. The method has been applied to the long-standing problem of texture determination in metallurgically compensated shaped-charge liners, and the first - though not yet complete - nondestructive measurement of texture as a function of depth in a shaped-charge liner has been achieved.

The neutron diffraction technique has also been employed for the first time to determine nondestructively, textural anisotropy in the matrix phase of tungsten-alloy KE penetrators. Because of the enormously greater x-ray scattering from tungsten relative to nickel or iron, this type of information is unobtainable either destructively or nondestructively except by means of neutrons.

Results obtained by means of small-angle neutron scattering have been outlined which demonstrate a capability which will be employed in the area of second-phase precipitates.

In summary, although neutron diffraction techniques for NDT are limited to laboratory environments, there are numerous examples where needed information cannot be obtained by any other means. In

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these cases the neutron techniques - since they are nondestructive - are seen as a means by which property/performance correlations can be determined and specifications established for routine inspection by other, more portable methods.

V. ACKNOWLEDGMENTS

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